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International application number: PCT/GB05/001290

International filing date: 04 April 2005 (04.04.2005)

Document type: Certified copy of priority document

Document details: Country/Office: GB
Number: 0407713.7
Filing date: 05 April 2004 (05.04.2004)

Date of receipt at the International Bureau: 02 May 2005 (02.05.2005)

Remark: Priority document submitted or transmitted to the International Bureau in compliance with Rule 17.1(a) or (b)

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2.	Patent application number *(The Patent Office will fill in this pa*	0407713.7
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	Patents ADP number *(if you know it)*	07649676002
	If the applicant is a corporate body, give country/state of its incorporation	UK
4.	Title of the invention	Mass Spectrometer
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MASS SPECTROMETER

The present invention relates to a mass spectrometer, and a method of mass spectrometry.

It is known to reduce the transmission of a continuous ion beam by a known factor using an Einzel lens and repetitively switching the Einzel lens back and forth between an attenuated and non-attenuated mode on a spectrum to spectrum basis. This method allows the dynamic range of a mass spectrometer to be increased. Specifically this patent discloses a method of attenuating the ion beam using an Einzel lens to defocus the ion beam so that the profile of the beam exceeds an aperture arranged downstream of the lens. In attenuated mode only a fraction of the ions pass through this aperture while the remaining ions strike the surrounding surfaces.

Defocusing or deflecting the ion beam, as described in the prior art, to reduce transmission of a continuous ion beam has the following disadvantages.

Firstly, it is difficult to predict the conditions to which the attenuation lens should be adjusted to result in the desired reduction in ion transmission. Generally such a system must first be calibrated by measuring the transmission of the system at several different lens conditions to empirically determine the relationship between the voltages applied to the attenuation lens and relative transmission of the system. This relationship may depend on the settings of other focussing elements in the system. Consequently, it may be necessary to recalibrate the attenuation lens at regular intervals to ensure an accurate estimation of the relative transmission.

Secondly, the portion of the ion beam which is not allowed to pass through the aperture and is incident on the surrounding surfaces may cause surface charging on or around the aperture plate. If this becomes significant this additional potential, in close proximity to the ion beam, will lead to a change in the focussing of the ion beam. This will lead to instability in the ratio between the attenuated and non-attenuated states.

Thirdly, the effect of the attenuation lens may cause a change in the cross-section profile, spatial and angular distributions, velocity or energy profile of the beam for which the subsequent mass analyser may have different performance, different mass resolution and different mass calibration.

Fourthly, if the cross section profile of the ion beam passing through the attenuation lens varies with mass to charge ratio (m/z), the relative transmission between the attenuated and non-attenuated modes may be different for different m/z values. This causes an additional complication in calibrating the effect of the attenuation lens across a wide range of m/z values. For example, the cross section profile of the ion beam exiting an electron impact (EI) ionisation or chemical ionisation (CI) source may vary with respect to mass to charge ratio due to the mass dispersing action of the stray magnetic field from magnets commonly employed to focus the ionising electron beam in the ionisation source. In a second example, an ion transfer device utilising RF voltages may have transmission and focussing properties which are dependent on m/z value.

It is therefore desired to provide an improved mass spectrometer. The preferred embodiment comprises a method of attenuating a continuous beam of ions by rapidly gating the transmission of the ions between zero transmission and high or full transmission. The degree of attenuation can be precisely controlled and predicted by varying the ratio between the zero and high or full transmission states (mark space ratio).

According to another embodiment a gas collision cell may be incorporated after the beam-gating device to disperse ions such that the beam is converted back to a continuous stream of ions.

In many mass spectrometric applications very complex mixtures of compounds are analysed. Individual components within these mixtures are present with a wide range of relative concentrations. This gives rise to a wide range of ion current intensities transmitted to the mass analyser and the ion detector. For many of these applications it is important to produce quantitative and qualitative data (in the form of exact mass measurement) for as many components as possible. This puts very high demands on the dynamic range of the ion source, mass analyser and detection system employed in the mass spectrometer. One method, which has been employed to extend the dynamic range for quantitative and qualitative analysis, is to adjust the transmission of the ion beam from the ion source to the mass analyser by a pre-determined factor. Data is then only recorded at a level of ion transmission, which is compatible with the mass spectrometer used. Transmission may be adjusted continuously based on an independent measurement of the ion current strength at regular

intervals during the analysis. In this method the ion beam transmission is continuously adjusted during the analysis so as to remain below the maximum level compatible with the mass spectrometer employed. Alternatively, the transmission may be repetitively switched between two predetermined transmission values during the analysis and data presented from either one or the other transmission mode dictated by the maximum ion current compatible with the mass spectrometer employed.

The most common way of controlling the relative transmission of an ion beam is to employ either a focusing or deflecting electrostatic lens in proximity to a restriction aperture downstream of this lens. The profile of the ion beam is made to increase by the action of the electrostatic lens or made to move in a direction orthogonal to the direction of the ion beam such that only a portion of the initial ion beam passes through the restriction aperture. The remaining ions are incident on the surface of the restricting aperture.

In the preferred embodiment the ion transmission is adjusted using a pulsed ion gate. For the period where the ion gate is closed no ions pass through the gate and the system has zero transmission. For the period where the gate is open a large proportion of the ions pass through the gate and the system has high or full transmission. By changing the mark space ratio between the two transmission modes the average flux of ions through the system may be adjusted.

This method of controlling transmission overcomes the problems associated with the conventional methods in that the factor by which the transmission of ions is reduced may be precisely controlled and predicted. The

relative transmission is directly proportional to the duty cycle of the gating pulse. This negates the requirement for calibration of the attenuating characteristics of the device.

Secondly, the gating device may be arranged so that during the zero transmission mode ions are directed away from the surfaces, which are in close proximity to the ion beam in the high or full transmission mode. This reduces the likelihood of surface charging in close proximity to the ion beam, which may effect focussing, and hence transmission, in the high or full transmission mode.

Thirdly, the ion beam is only transmitted under high or full transmission conditions. Under these conditions the gating device is effectively inactive. Thus the overall transmission may be reduced without introduction of the significant spatial aberrations or energy spread of the ion beam associated with the prior art.

Fourthly, because the ion beam is only transmitted under high or full transmission conditions, where the gating device is inactive, the method disclosed will result in a constant attenuation factor with respect to m/z value even if the ion beam is inhomogeneous with respect to m/z value.

Various embodiments of the present invention will now be described, by way of example only, and with reference to the accompanying drawings in which:

Fig. 1 shows a schematic representation of a conventional Einzel lens arrangement wherein the voltages applied to the lens elements shown are such that the ion beam is at full transmission;

Fig. 2 shows the same arrangement as shown in Fig. 1 wherein the transmission of an ion beam is reduced due to the defocusing effect of the electrodes;

Fig. 3 shows a second conventional method of reducing transmission involving deflection of the ion beam;

Fig. 4 shows a first embodiment of the invention wherein the voltages, applied to the lens elements shown, are such that the beam is at full transmission. This state represents the high or full transmission cycle of the ion gate;

Fig. 5 shows the same embodiment as shown in Fig. 4 wherein the voltages applied to the lens elements shown are such that the beam is at zero transmission. This state represents the zero transmission cycle of the ion gate;

Fig. 6 shows a second method of gating the ion beam transmission to zero using a deflection voltage;

Fig. 7 shows a timing diagram representing the voltage pulse applied to the ion gate shown in Figs. 3-5. In this example the duty cycle is 10:1. This would result in a relative transmission of 10%;

Fig. 8 shows a schematic representation of the effect of the repetitive pulsing of an ion gate on the transmission of the ion beam during operation of the preferred embodiment;

Fig. 9 shows a preferred embodiment of the invention wherein the ion gate shown in Fig. 8 is shown preceding an RF-only ion transfer device operating in the microbar to millibar pressure regime;

Fig. 10 shows a SIMION (RTM) model of the preferred embodiment of the invention wherein the ion gate is OFF and the transmission of ions is high;

Fig. 11 shows a 3D potential energy diagram of the SIMION (RTM) model shown in Fig. 10;

Fig. 12 shows a SIMION (RTM) model of the preferred embodiment of the invention wherein the ion gate voltage is ON and the transmission of ions is zero;

Fig. 13 shows a 3D potential energy diagram of the SIMION (RTM) model shown in Fig. 12;

Fig. 14 shows an experimentally determined graph of relative transmission against the duty cycle of the pulsed ion gate using the preferred embodiment;

Fig. 15 shows the same data as shown in Fig. 14 plotted on a log-log scale for clarity;

Fig. 16A shows an example of an ESI mass spectrum at full transmission, and

Fig. 16B shows the same analysis as shown in Fig. 16A at 10% transmission according to the preferred embodiment;

Fig. 17 shows a narrow m/z region from the mass spectrum in Fig 16A at full transmission; and

Fig. 18 shows a narrow m/z region from the mass spectrum shown in 16B at 10% transmission.

Figs. 1-3 show a conventional arrangement. Referring to Fig. 1 a beam of positive ions 1 is shown traversing an electrostatic lens assembly represented by electrodes 2,3,4 and exit slit 5. The electrodes 2,3,4 are held at nominally identical voltages providing an essentially field free region. The ion beam shown is fully transmitted through the exit slit 5. In Fig. 2 the middle electrode 3 has been raised to a voltage above the voltage applied to the first and third electrodes 2,4 and the exit slit 5. The ion beam is defocused and only a portion of the ion beam passes through the exit slit 5. In this state the ion

transmission is reduced by a fixed amount. The remainder of the ion beam is incident upon the surface of the exit slit 5 in the proximity of the slit opening.

Fig. 3 shows an alternative arrangement. In this case electrode 3a is raised to a voltage above the voltage applied to electrodes 2,3b,4 and exit slit 5. The ion beam is deflected away from electrode 3a and only a portion of the ion beam is transmitted through the exit slit 5. In this state the ion transmission is reduced by a fixed amount. The remainder of the ion beam is incident on the surface of the exit slit in the proximity of the slit opening.

According to a first embodiment of the present invention as shown in Fig. 4, a beam of positive ions 1 traverses an electrostatic lens assembly represented by electrodes 2,3,4 and exit slit 5. At a time T1 the electrodes are held at potentials such that the ion beam is fully transmitted through the exit slit 5. At a time T2 as shown in Fig. 5 a gate voltage capable of retarding the entire beam of ions is preferably applied to the third electrode 4. As a result the ions are accelerated in the opposite direction to the direction of the initial ion beam and are incident on the surface of the second electrode 3. The ion beam is shown with zero transmission through the exit slit 5.

In this embodiment, referring to Fig. 7, the gate voltage is applied to the third electrode 4 for a duration ΔT1. During this time the transmission of the ion beam through the exit slit 5 is zero. At the end of this time the gate voltage is switched OFF for a duration ΔT2. During this time the transmission of the ion beam through the exit slit 5 is high. This sequence

is repeated as indicated by the voltage-timing diagram Fig. 7.

The beam of ions passing through the exit slit 5 consists of discrete ion packets. The mark space ratio of the pulsed transmission gate is given by the equation $\Delta T2/\Delta T1$. The average relative transmission of the ion beam is proportional to the duty cycle of the pulsed ion gate $\Delta T2/(\Delta T1+\Delta T2)$. In the voltage-timing diagram shown in Fig. 7 the duty cycle is 0.1. The transmission is 10% of the original ion transmission.

Fig. 6 shows an alternative embodiment wherein the ion gate consists of a deflection electrode 3a. For time duration $\Delta T1$ a deflection voltage is applied to electrode 3a such that the ion beam 1 is incident on the third electrode 4 and transmission through electrode 5 is zero. For time duration $\Delta T2$ the deflection voltage is OFF and the transmission of the ion beam through the exit electrode 5 is high.

Fig. 8 shows a generalised representation of a preferred pulsed ion gate in operation. A continuous beam of ions 1 passes through ion gate 2, which is pulsed, between high or full transmission and zero transmission with a preset duty cycle. The resultant ion beam 3 appears as discrete packets of ions with an average transmission lower than the original continuous ion beam.

With reference to Fig. 9, in a preferred embodiment a continuous beam of ions 1 passes through ion gate 2, which is pulsed between high or full transmission and zero transmission with a preset duty cycle. In the preferred embodiment the total cycle time for the zero and high or full transmission states of the ion gate is

in the order of 100 - 1000 μs although the cycle time could be much longer. The resultant ion beam 3 appears as discrete packets of ions with an average transmission lower than the original continuous ion beam. This discontinuous ion beam then enters an RF-only transfer device operating in the microbar to millibar pressure regime. Within this device sequential collisions between the ions and the background gas result in the internal energy of the ions being lowered to approach that of the background gas (collisional damping). The initial velocity of the ions entering the ion guide is damped and the ions collapse towards the central axis. As the ions become thermalised, their motion becomes more random. This has the effect of dispersing the spatial distribution of the discrete packets of ions along the axis of the ion guide. Hence a continuous stream of ions is converted into an attenuated continuous stream of ions in which the degree of attenuation is controlled by controlling the duty cycle of the pulsed transmission gate.
This coupling of a pulsed attenuation lens preceding a collisional damping ion guide has several advantages in application to mass spectrometry.

Firstly, the collisional damping ion guide effectively decouples the pulsed attenuation lens from the rest of the spectrometer. Any spatial spreading and/or energy spread induced by the action of the pulsed ion gate will not be apparent in the continuous ion beam exiting the ion guide. This feature is important if the device is coupled to an orthogonal time of flight (TOF) mass spectrometer. The ion beam may be attenuated by a precise amount using the pulsed ion gate without affecting the performance of an oa-TOF mass analyzer in

terms of mass resolution mass calibration and mass accuracy.

Secondly, the ion beam transmitted to the mass analyser will appear as an attenuated continuous beam rather that as discrete ion packets with a lower average ion flux. This feature is very useful when coupling the device to an orthogonal time of flight mass spectrometer.

Without a collisional damping ion guide after the pulsed ion gate the resultant ion beam within the orthogonal acceleration region of the analyzer may retain some of the discrete nature of the ion packets produced by the ion gate alone. Depending on the frequency at which the ion gate is pulsed, the time of flight between the ion gate and the orthogonal acceleration region, and the frequency of the orthogonal acceleration pulse this may result in some mass discrimination in the final mass spectrum produced.

Thirdly, if the ion beam flux is not continuous but fluctuates between a high and low flux, the full advantage of increased dynamic range will not be realised.

Fig. 10 shows a SIMION (RTM) model of the preferred embodiment. In this figure the ion gate is open and the transmission of ions into an RF-only collisional damping transfer device is high. A beam of positive ions 1 at 3eV of axial energy is shown exiting a low pressure RF-only hexapole ion guide 2 held at a relative potential of 0V. Electrodes 3a and 3b are held at a relative potential of -57V. Electrode 4 is held at -2V, electrode 5 at -1V and the entrance to an RF-only transfer device 6 operating at a pressure of $9x10^{-3}$ mbar is held at -2V. Ions are focussed near to or upstream

from the entrance to the transfer device 6 and transmission through this device is high.

Fig. 11 shows a three-dimensional potential energy diagram of the embodiment shown in Fig 10.

Fig. 12 shows a SIMION (RTM) model of the preferred embodiment. In this figure the ion gate is closed and the transmission of ions into an RF-only collisional damping transfer device is zero. A beam of positive ions 1 at 3eV of axial energy is shown exiting an RF-only hexapole ion guide 2 held at a relative potential of 0V. Electrode 3a is held at a relative potential of -47V and electrode 3b at -67V. Electrode 4 is held at +8V, electrode 5 at -1V and the entrance to the high pressure RF-only transfer device 6 at -2V. Ions are accelerated and deflected by electrodes 3a and 3b, and are then retarded by electrode 4. Ions are then reaccelerated towards the rear surface of electrode 4 ensuring that none of the ions return into the RF-only region 2. The transmission of the arrangement is zero.

Fig. 13 shows a three-dimensional potential energy diagram of the embodiment shown in Fig 12.

Fig. 14 shows an experimentally determined graph showing the relationship between the observed relative transmission of the ion beam and the duty cycle of the pulsed ion gate using the preferred embodiment. It can be seen that there is a direct and predictable relationship between the relative transmission and the duty cycle of the pulsed ion gate. For clarity this data is shown in Fig. 15 re-plotted using log of the relative transmission against log of the duty cycle of the pulsed ion gate. The total cycle time for the ion gate ON and OFF pulse was 300 μs for this experiment.

Fig. 16A shows an electrospray MS-MS orthogonal time of flight spectrum from an infusion of (Glu)-fibrinopeptide-B m/z 785.8, acquired at full transmission. Ten mass spectra, each of 1.2 seconds duration, were averaged in this case. Fig. 16B shows a mass spectrum acquired according to the preferred embodiment after attenuating the ion beam by 90% using the pulsed ion gate preceding a collisional damping transfer device downstream of a time of flight mass analyser. The gate was pulsed with a duty cycle of 0.1 and a total cycle time of 300 µs. 100 mass spectra, of 1.2 seconds duration, were averaged to produce this mass spectrum. It can be seen that the amount of attenuation is constant for peaks over the entire mass range shown. The measured attenuation factor, based on the intensity of the peak at m/z 684.35, is 89.9%.

Fig. 17 shows a narrow m/z range from the same mass spectrum shown in Fig. 16A at full transmission. Fig. 18 shows a narrow m/z range from the same mass spectrum shown in Fig. 16B but at 10% transmission. No significant effect on peak resolution peak shape and m/z position is evident due to the action of the pulsed ion gate. This illustrates that the pulsed ion gate is effectively decoupled from the mass analyser by the presence of the RF-only ion guide operated at microbar to millibar pressures.

It will be clear to anyone skilled in the art that there are many other methods of rapidly pulsing an ion beam between zero transmission and high or full transmission. These methods include electrostatic, magnetic and mechanical methods.

In addition, it is not necessary to reduce the transmission to zero during the low transmission cycle

of the pulsed ion gate to attain a degree of attenuation using this method. The transmission may instead be reduced to some preset value between zero transmission and full transmission. However, using the pulsed gate in this way may result in a greater likelihood of surface charging on the electrodes used. This in turn may lead to instability in the attenuation factor. Similarly, it may not be possible to predict the attenuation factor directly from the duty cycle if the transmission is not reduced to zero.

The preferred embodiment described may be used to provide controlled attenuation of a continuous ion beam to be mass analysed using the following mass analysers: orthogonal time of flight, axial time of flight, 3D ion traps, linear ion traps, fourier transform ion cyclotron resonance mass spectrometer (FTICR), magnetic sector mass spectrometer, quadrupole mass spectrometer. In addition the ion beam passing through the device may be subject to MS or MSMS or MS^n analysis using any combination of the above.

The preferred embodiment described may be used to provide controlled attenuation of a continuous ion beam, transmission of the ion beam to be mass analysed using the following continuous or quasi continuous ion sources: ESI, APPI, APCI, MALDI, LDI, APMALDI, DIOS, EI, CI, FI, FD, ICP, FAB, LSIMS.

The method described may be used as part of an automatic ion beam transmission control during analysis. In this application a measurement of the ion current is made at regular intervals during the analysis. The amount of attenuation required is then repeatedly calculated from this measurement as the analysis proceeds. The measurement of ion current may be made by

examination of the mass spectral data, recorded as the analysis proceeds. The total ion current recorded or the ion current at a selected mass or masses can then be used to determine an attenuation factor for the next mass spectrum recorded.

Alternatively, using the method disclosed, during the period of time that the pulsed ion gate is closed and transmission is zero, ions may be directed towards a separate ion detector in the proximity of the ion gate. The signal recorded using this detector may be used to calculate the total ion current at the ion gate based on the duty cycle. This measurement may then be used to calculate a new duty cycle for the gate if the ion current exceeds the level, which can be accommodated by the mass analyser or detector employed. For instance, this method provides a means of automatically reducing the number of ions, per unit time, entering an ion trap mass analyser based on the known maximum number of ions.

In the preferred embodiment the pulsed ion gate precedes an RF-only transfer device operated in the microbar to millibar pressure regime. This may be a multipole transfer lens, a segmented multipole or stacked ring ion tunnel or stacked ring ion funnel. The device may utilise a linear acceleration field or a travelling wave method to propel ions through the device. This may also be used to ensure that ions are resident in the transfer device for a total time applicable to the particular mode of operation of the pulsed ion gate.

Although the present invention has been described with reference to preferred embodiments, it will be understood by those skilled in the art that various changes in form and detail may be made without departing

from the scope of the invention as set forth in the accompanying claims.

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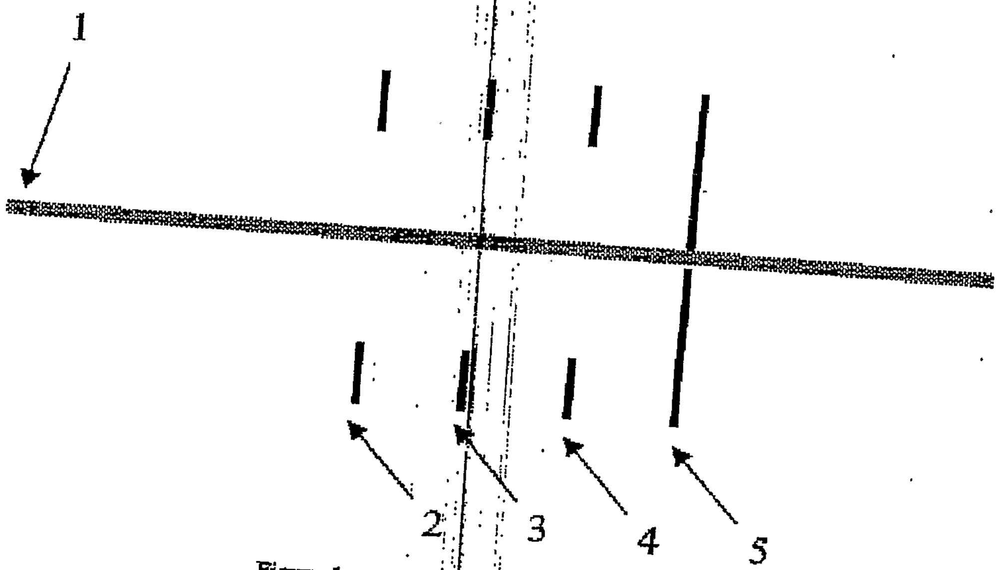

Figure 1

PRIOR ART

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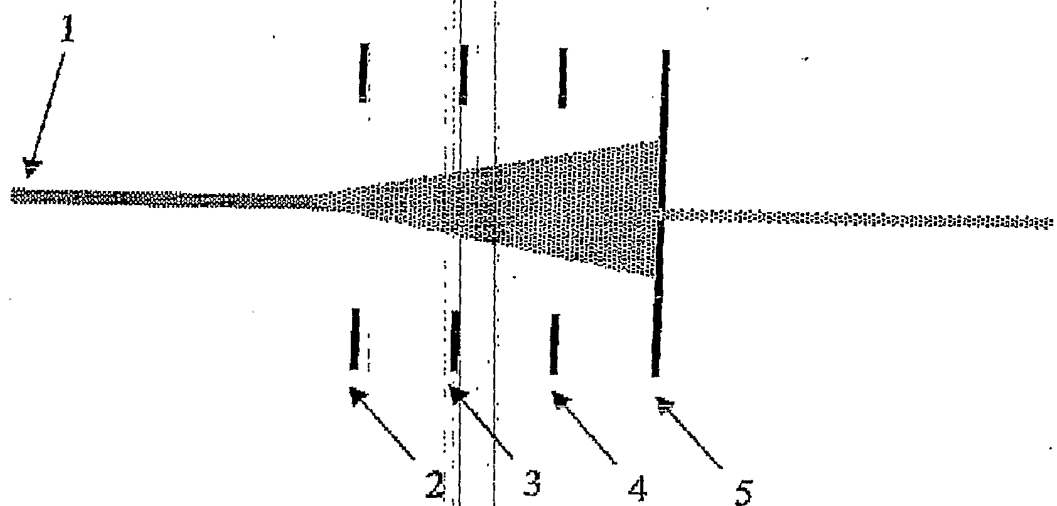

Figure 2

PRIOR ART

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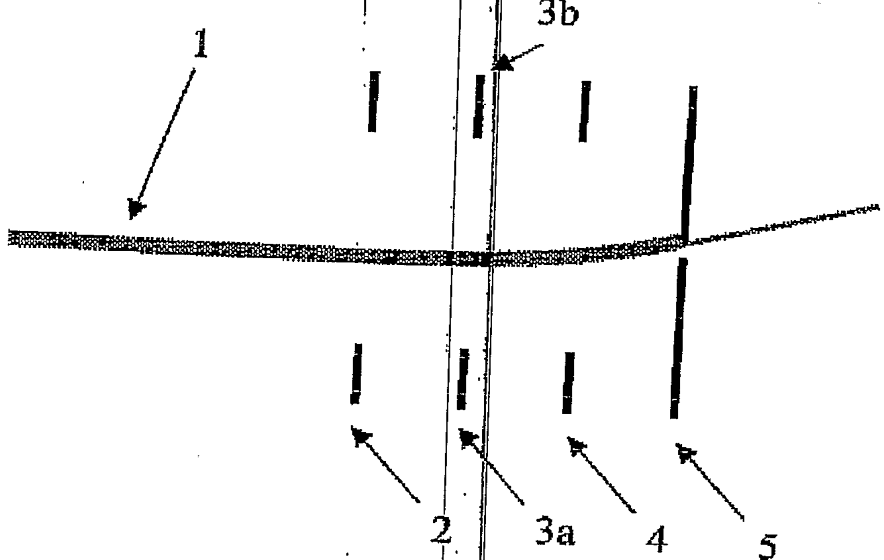

Figure 3

PRIOR ART

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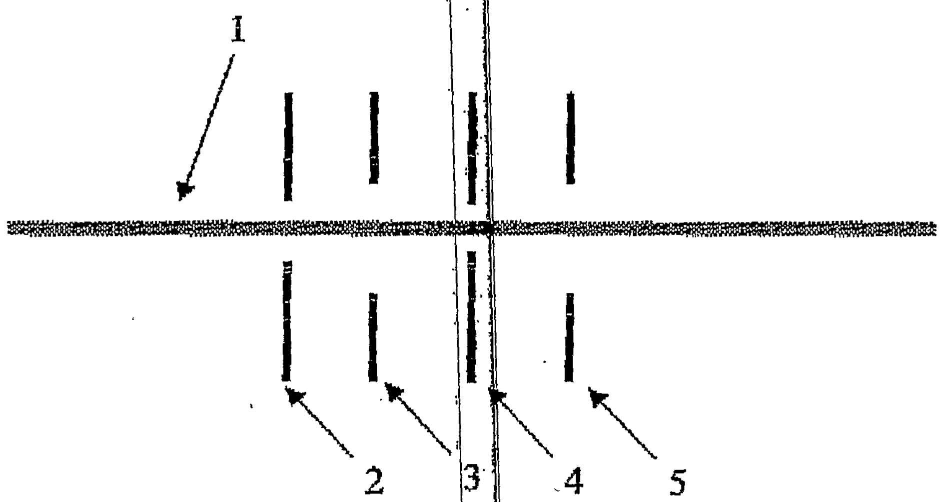

Figure 4

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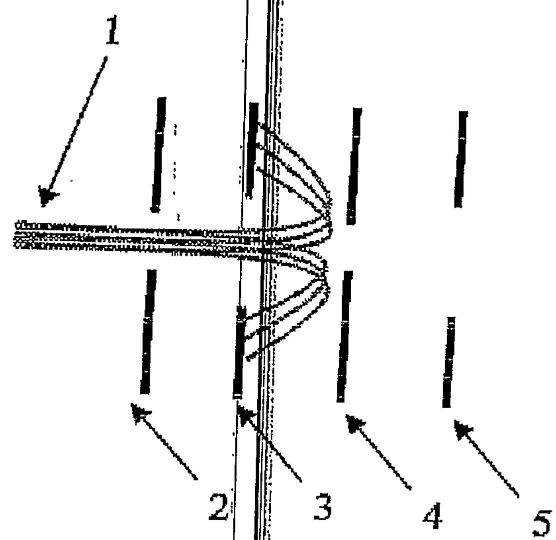

Figure 5

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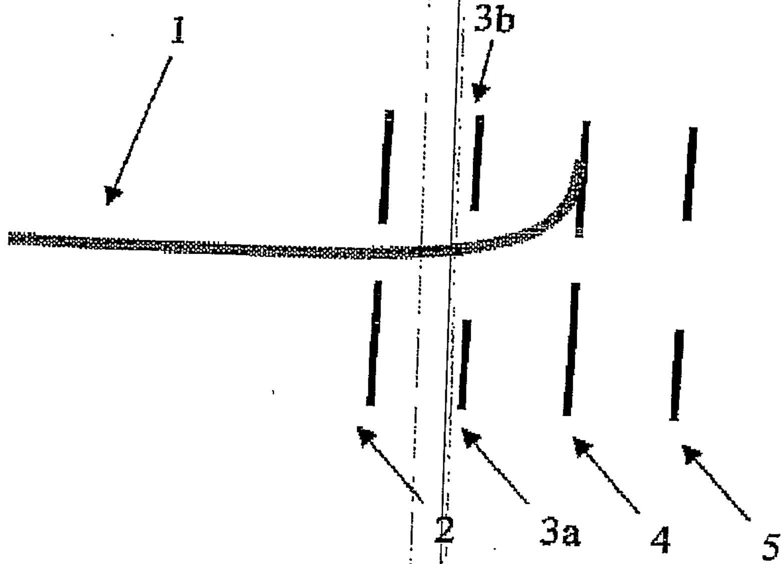

Figure 6

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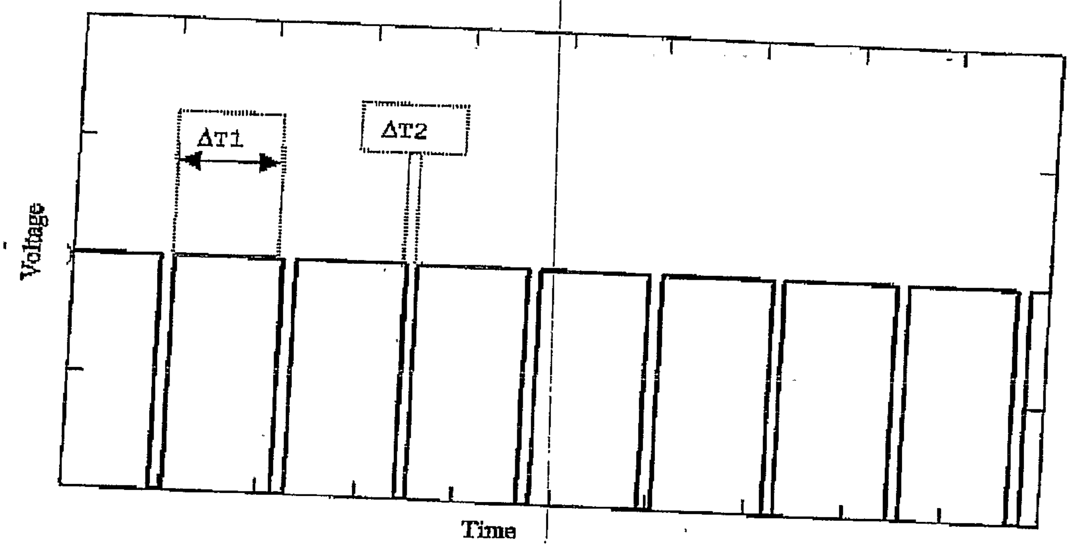

Figure 7

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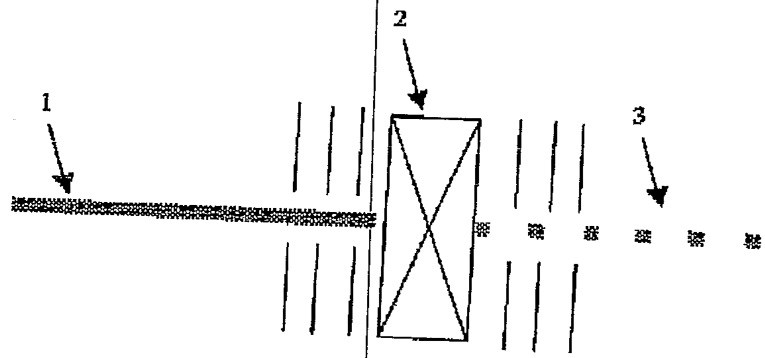

Figure 8

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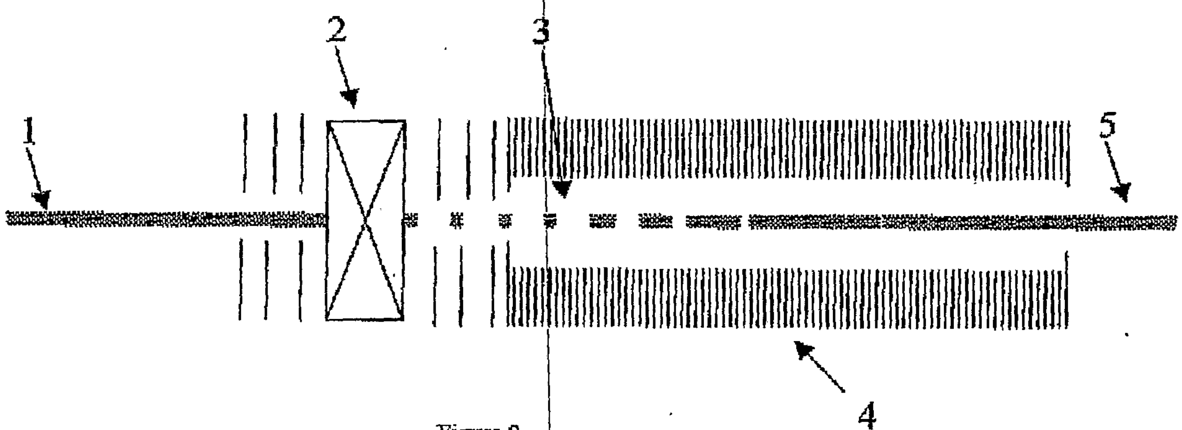

Figure 9

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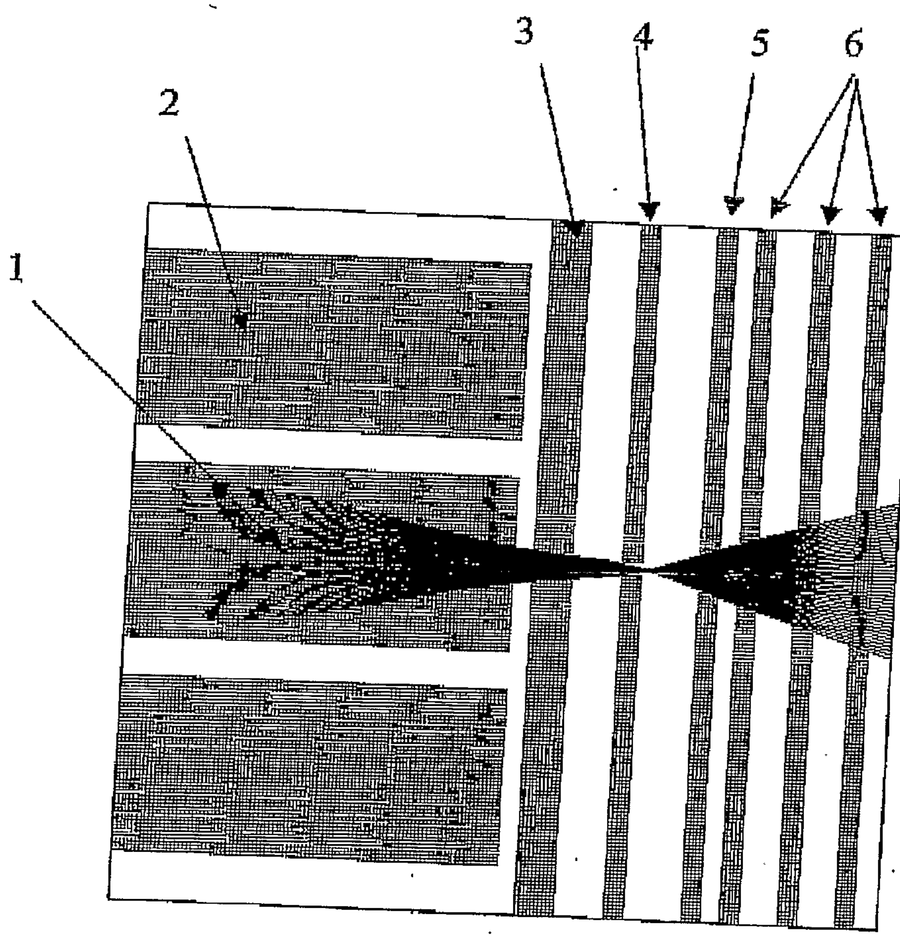

Figure 10

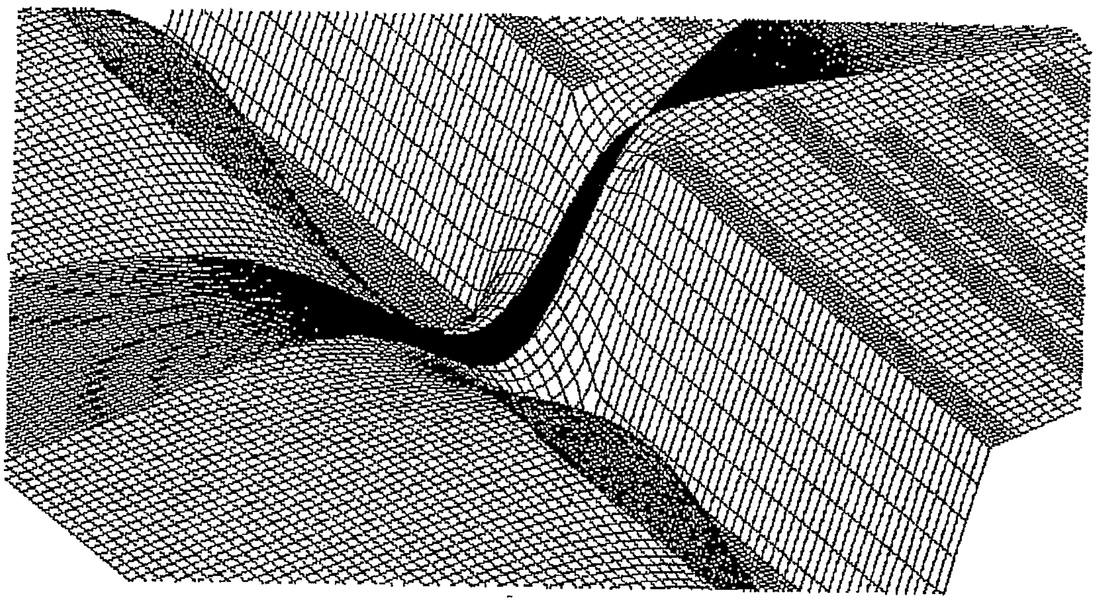

Figure 11

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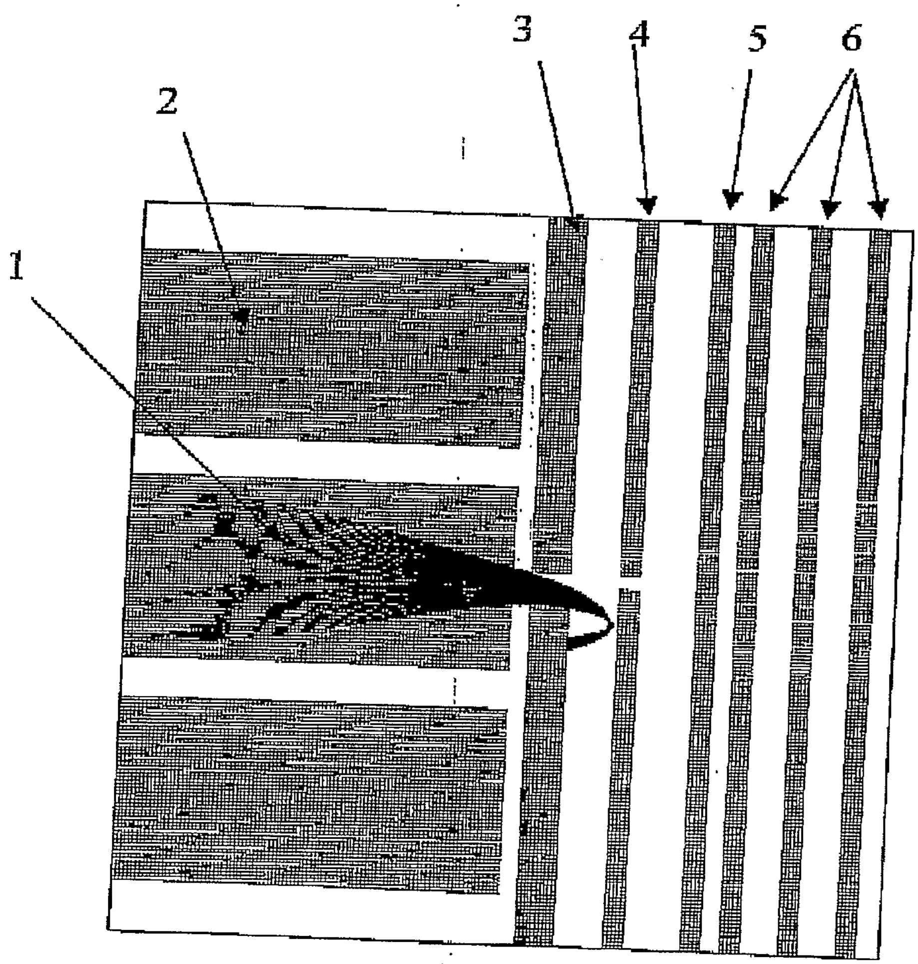

Figure 12

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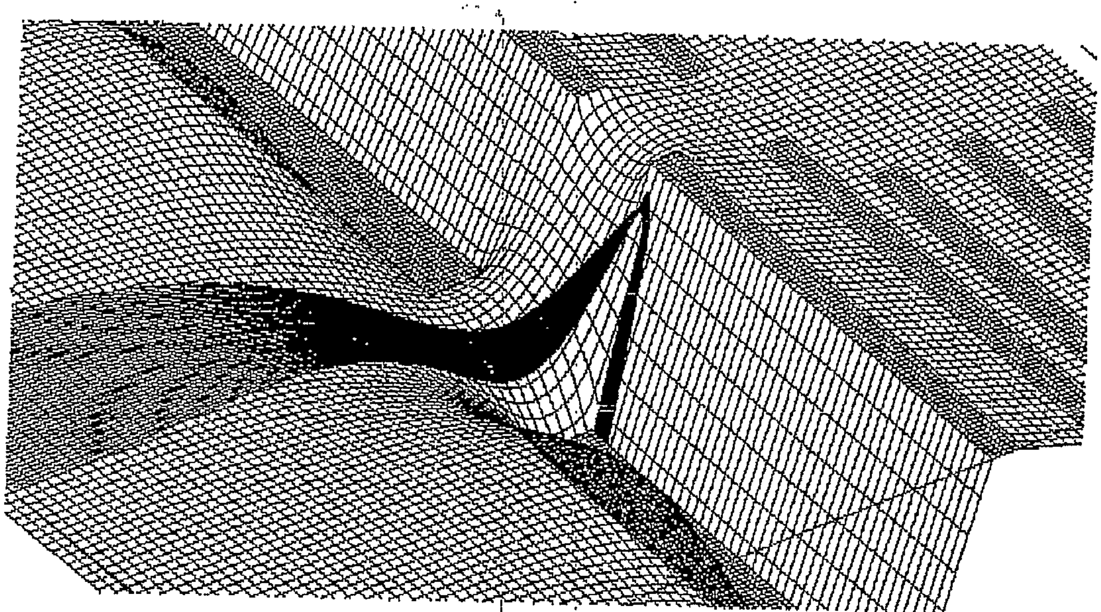

Figure 13

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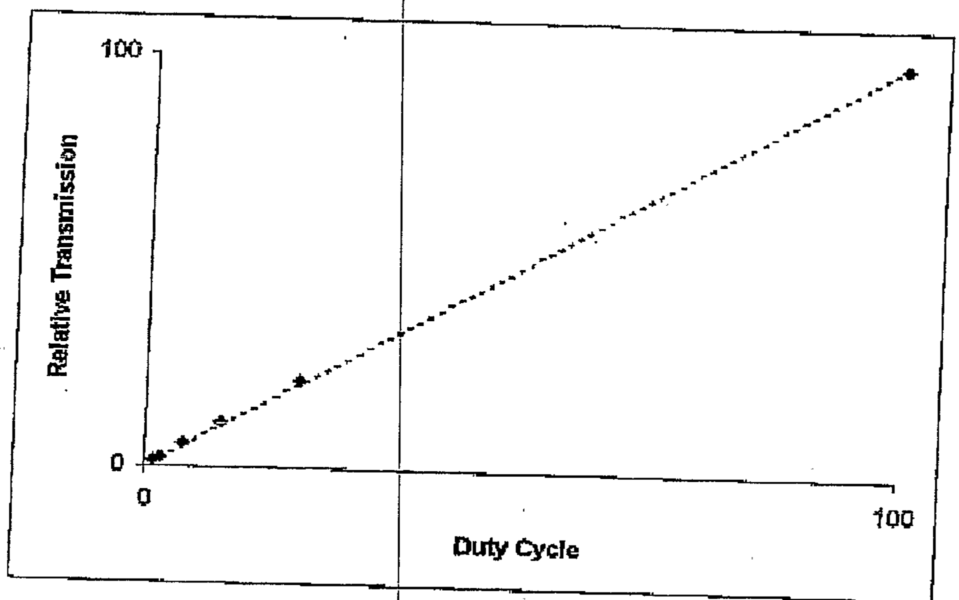

Figure 14

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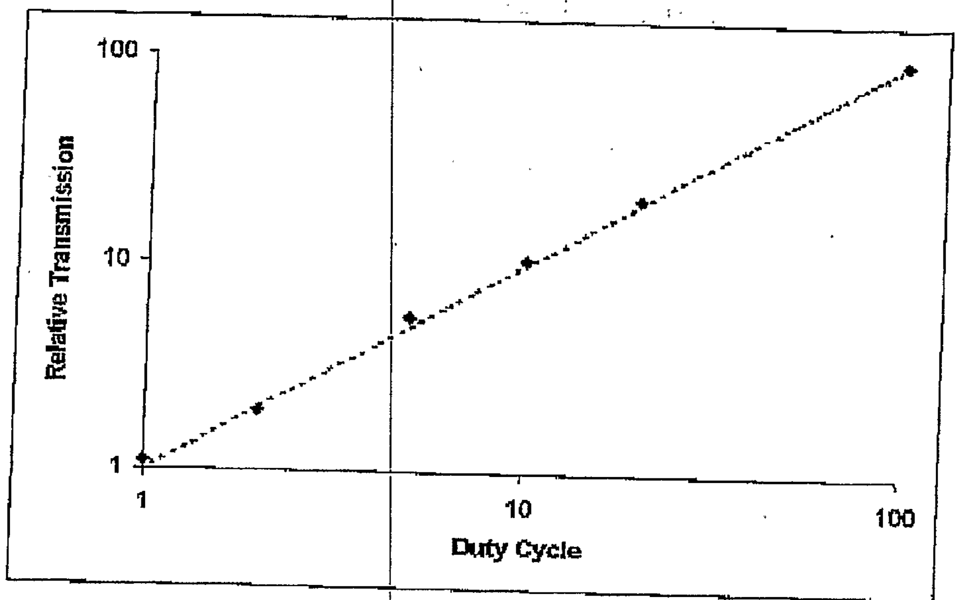

Figure 15

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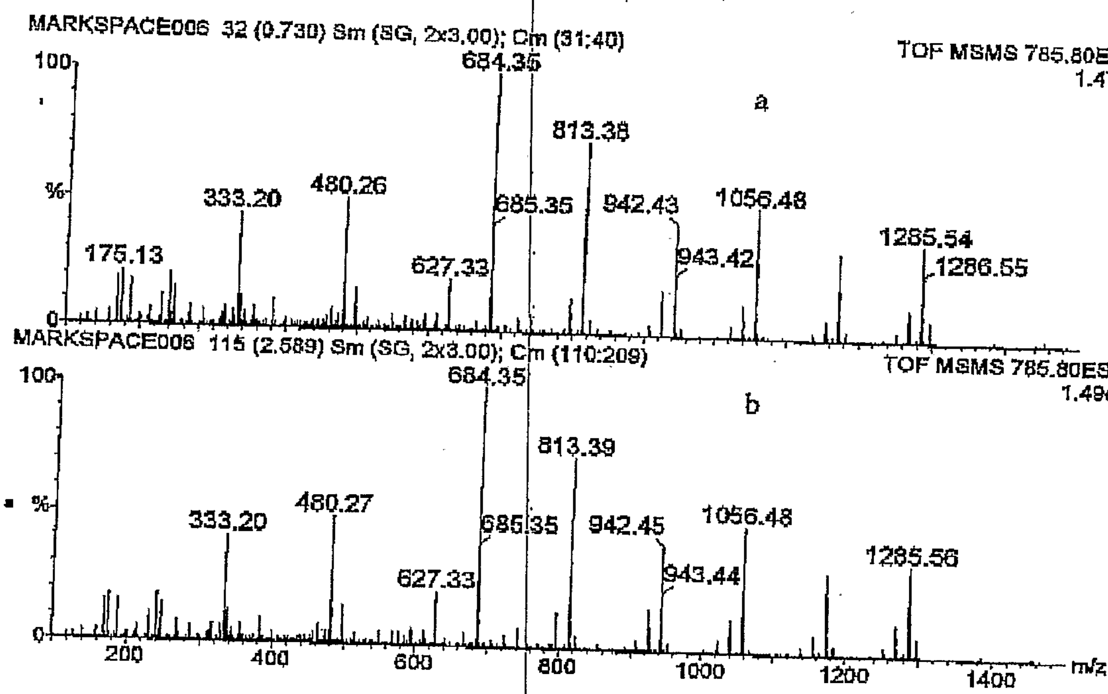

Figure 16

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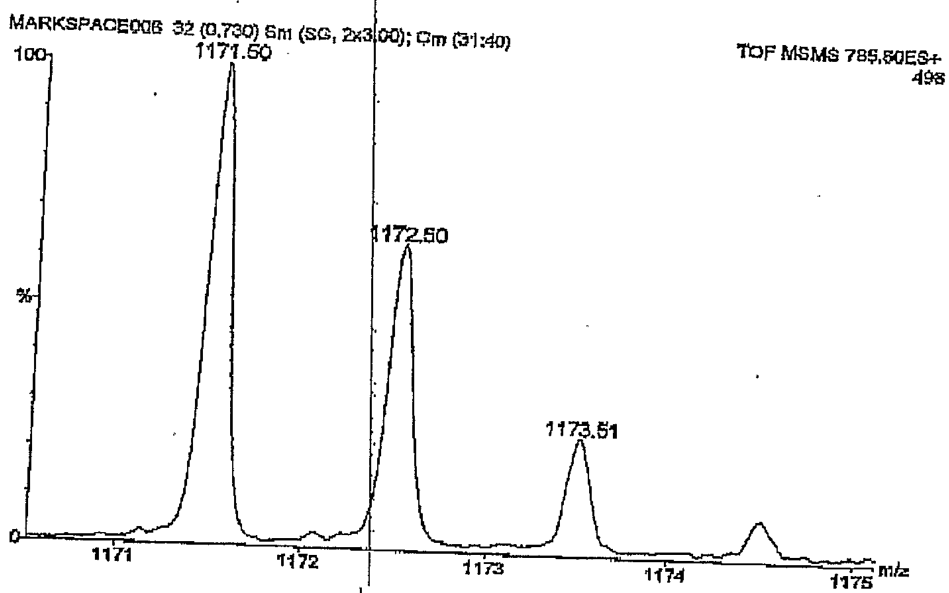

Figure 17

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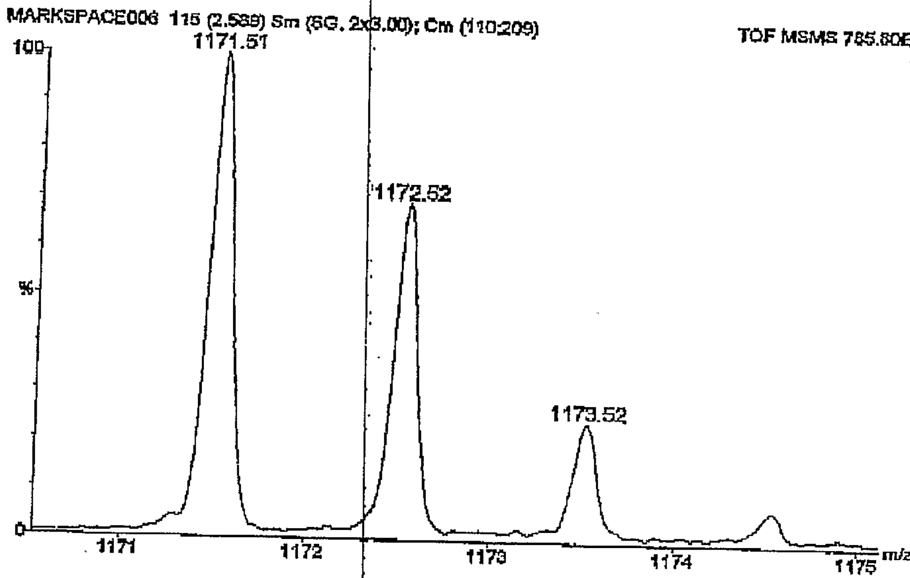

Figure 18